

JC07 Rec'd PCT/PTO 22 JAN 2002

FORM PTO-1390 (REV. 1-98)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER 0517-1001	
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371				U.S. APPLICATION NO. (If known, see 37 CFR 1.5 <b>10/031484</b>	
INTERNATIONAL APPLICATION NO. PCT/FR00/02106		INTERNATIONAL FILING DATE July 21, 2000		PRIORITY DATE CLAIMED July 21, 1999	
TITLE OF INVENTION METHOD FOR MAKING EMULSIONS AND IMPLEMENTING DEVICE					
APPLICANT(S) FOR DO/EO/US Eric FERRET, Pierre-Andre MARECHAL, Patrick GERVAIS, Jean-Marie PERRIER-CORNET, Philippe MARIE, Isabelle DE LAMARLIERE, Elisabeth LUSTRAT & Gilles FRANCH					
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:					
1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. 2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. 3. <input checked="" type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1). 4. <input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date. 5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2)) a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau). b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau. (see PCT/IB/308) c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). 6. <input checked="" type="checkbox"/> A translation of the International Application into English (35 U.S.C. 371(c)(2)). 7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)) a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau). b. <input type="checkbox"/> have been transmitted by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input type="checkbox"/> have not been made and will not be made. 8. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)). 9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). 10. <input type="checkbox"/> A translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). Items 11. to 16. below concern document(s) or information included: 11. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98. 12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. 13. <input checked="" type="checkbox"/> A FIRST preliminary amendment. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. 14. <input type="checkbox"/> A substitute specification. 15. <input type="checkbox"/> A change of power of attorney and/or address letter. 16. <input checked="" type="checkbox"/> Other items or information:  Form PCT/IB/308./ International Preliminary Examination Report. Abstract. Search Report. Application Data Sheet.					

U.S. APPLICATION NO. (if known, see 37 CFR 1.5)		INTERNATIONAL APPLICATION NO.		ATTORNEY'S DOCKET NUMBER	
10/031484		PCT/FR00/02106		0517-1001	
<p>17. <input checked="" type="checkbox"/> The following fees are submitted:</p> <p><b>BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):</b></p> <p>Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... \$1,040.</p> <p>International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... 890.</p> <p>International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... 740.</p> <p>International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... 710.</p> <p>International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) ..... 100.</p> <p style="text-align: center;"><b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b></p>				CALCULATIONS PTO USE ONLY	
				\$ 890	
				\$ 130	
				\$ 0	
				\$ 0	
<p>Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).</p>				\$ 130	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$	
Total claims	10 - 20 =	0	x \$ 18.	\$ 0	
Independent claims	3 - 3 =	0	x 84.	\$ 0	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)				\$ + 280.	
<b>TOTAL OF ABOVE CALCULATIONS =</b>				\$ 1020	
Reduction of 1/2 for small entity				\$	
<b>SUBTOTAL =</b>				\$ 1020	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$	
<b>TOTAL NATIONAL FEE =</b>				\$ 1020	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +				\$	
<b>TOTAL FEES ENCLOSED =</b>				\$ 1020	
				Amount to be refunded: \$	
				charged: \$	

- a. ☒ A check in the amount of \$ 1,020 to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. \_\_\_\_\_ in the amount of \$ \_\_\_\_\_ to cover the above fees. A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required by 37 CFR 1.16 and 1.17, or credit any overpayment to Deposit Account No. 25-0120. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO: January 22, 2002

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10/031484

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VERIFICATION OF A TRANSLATION

I, DIANA MARY UNDERWOOD, MIL, MITI, the below-named translator, hereby declare that:

My name and post office address are as stated below.

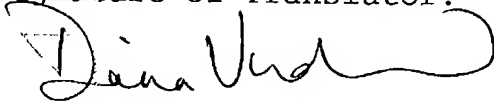
That I am knowledgeable in the English language and in that language in which the below identified International Application was filed, and that I believe the English translation of International Application No. PCT/FR00/02106 is a true and complete translation of the above-identified International Application as filed.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the Application or any Patent issued thereon.

Date: 11<sup>th</sup> January 2002

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Application Data Sheet

**Application Information**

Application Type:: Regular  
Subject Matter:: Utility  
Suggested Classification::  
Suggested Group Art Unit::  
CD-ROM or CD-R?:: None  
Number of CD disks::  
Number of Copies of CDs::  
Sequence Submission?:: None  
Computer Readable Form (CRF):: No  
Number of copies of CRF:: 0  
Title:: METHOD FOR MAKING EMULSIONS AND  
IMPLEMENTING DEVICE  
Attorney Docket Number:: 0517-1001  
Request for Early Publication?:: No  
Request for Non-Publication?:: No  
Suggested Drawing Figure::  
Total Drawing Sheets:: 6  
Small Entity?:: No  
Latin Name::  
Variety Denomination Name::  
Petition Included?:: No  
Petition Type::  
Licensed US Gov't Agency::  
Contract or Grant Numbers::  
Secrecy Order in Parent No  
Appl.?::

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**Representative Information**

Representative Customer Number::	000466
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**Domestic Priority Information**

Application::	Continuity Type::	Parent Application::	Parent Filing Date::
This application	National Stage of	PCT/FR00/02106	7/21/00

**Foreign Priority Information**

Country::	Application Number::	Filing Date::	Priority Claimed::
FRANCE	99/09448	7/21/99	Yes

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State or Province of Mailing Address::

Country of Mailing Address::

Postal or Zip Code of Mailing Address::

100331484 10/03/02 1484

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PATENTS

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Eric FERRET et al.

Serial No. (unknown)

Filed herewith

METHOD FOR MAKING EMULSIONS  
AND IMPLEMENTING DEVICE

PRELIMINARY AMENDMENT

Commissioner for Patents

Washington, D.C. 20231

Sir:

Prior to calculation of the filing fee, please substitute claims 1-10 as originally filed, which appear on pages 15-17, with claims 1-10 as filed in the Article 34 amendment of September 11, 2001. The pages containing claims 1-10 are marked "AMENDED SHEETS" and are attached hereto. Following the insertion of these claims, please amend as follows:

IN THE CLAIMS:

Amend claim 3 as follows:

--3. (amended) Method according to claim 1, characterised in that the temperature of the dispersed phase under pressure is regulated in a temperature range included between -20°C and +80°C.

Amend claim 4 as follows:

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--4. (amended) Method according to claim 1, characterised in that the contacting of the resulting coherent jet (9) with the continuous phase is obtained by positioning said resulting coherent jet (9) in immersion in the continuous phase in static or virtually static position.

Amend claim 5 as follows:

--5. (amended) Method according to claim 1, characterised in that the contacting of the resulting coherent jet (9) with the continuous phase is obtained thanks to means ensuring introduction of the continuous phase in said resulting coherent jet (9) and simultaneously their emulsion which in that case constitutes a final coherent jet (13).

Amend claim 8 as follows:

--8. (amended) Emulsifier device according to claim 6, characterised in that the means (3) for connection between the high-pressure pump (2) and the spray means (4) are provided with temperature regulating means (6) over all or part of their length.

Amend claim 10 as follows:

--10. (amended) Emulsifier device according to claim 6, characterised in that the drawing-off means (10) are provided with temperature regulation means (33, 36, 37, 38, 39) connected to the heat exchanger (13).--

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R E M A R K S

The above changes in the claims merely place the national phase application in the same condition as it was during Chapter II of the international phase, with the multiple dependencies being removed.

Attached hereto is a marked-up version of the changes made by the current amendment. The attached page is captioned "VERSION WITH MARKINGS TO SHOW CHANGES MADE".

Respectfully submitted,

YOUNG & THOMPSON

By

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January 22, 2002

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## CLAIMS

## AMENDED SHEETS

1 - Method of continuously or discontinuously making a mixture or an emulsion from at least one emulsifying agent and at least two fluids known to be immiscible, for example a liquid fat mixed with water and an appropriate emulsifying agent, said fluids defining a dispersed phase and a continuous phase, characterised in that, the dispersed phase being either contained in an adapted tank or continuously supplied, at least the following steps are carried out in order:

- 5 - the dispersed phase is pressurised by conventional high-pressure pumping means, then
- the dispersed phase is suddenly depressurised using means generating a coherent jet (5) then
- an appropriate emulsifying agent is then introduced into said coherent jet (5) thanks to means ensuring mixing of the dispersed phase with said emulsifying agent and thus procuring a resulting coherent jet (9) then
- said resulting coherent jet (9) is contacted with the continuous phase in order finally to obtain the emulsion.

2 - Method according to Claim 1, characterised in that the fluid or fluids forming the dispersed phase is pressurised at a pressure greater than or equal to 200 MPa.

3 - Method according to either one of the preceding Claims, characterised in that the temperature of the dispersed phase under pressure is regulated in a temperature range included between -20°C and +80°C.

4 - Method according to any one of the preceding Claims, characterised in that the contacting of the resulting coherent jet (9) with the continuous phase is obtained by positioning said resulting coherent jet (9) in immersion in the continuous phase in static or virtually static position.

5 - Method according to any one of Claims 1 to 4, characterised in that the contacting of the resulting

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coherent jet (9) with the continuous phase is obtained thanks to means ensuring introduction of the continuous phase in said resulting coherent jet (9) and simultaneously their emulsion which in that case constitutes a final coherent jet (13).

6 - Device for continuously or discontinuously making a mixture or an emulsion from at least one emulsifying agent and at least two fluids known to be immiscible, for example a liquid fat mixed with water and an appropriate emulsifying agent, said fluids defining a dispersed phase and a continuous phase, implementing the method according to Claim 4 and comprising a high-pressure pump (2) of which the inlet is connected to a source of fluid such as a tank (1) containing a dispersed phase, characterised in that the outlet of the high-pressure pump (2) is connected, by connection means (6), to means (4) for spraying the dispersed phase in the form of a coherent jet (5) co-operating with means (7), connected to an open tank (8) and using the Venturi effect, for introduction of the emulsifying agent in said coherent jet (5) to form a resulting coherent jet (9) in a focussing tube (30) fast with the introduction means (7) and opening out, in immersion, in the continuous phase contained in means (10) for drawing off the emulsion continuously or discontinuously.

7 - Device for continuously or discontinuously making a mixture or an emulsion from at least one additive and at least two fluids known to be immiscible, for example a liquid fat mixed with water and an appropriate emulsifying agent, said fluids defining a dispersed phase and a continuous phase, implementing the method according to Claim 5 and comprising a high-pressure pump (2) of which the inlet is connected to a source of fluid such as a tank (1) containing a dispersed phase, characterised in that the outlet of the high-pressure pump (2) is connected, by connection means (3), to means (4) for spraying the dispersed phase in the form of a coherent jet (5), provided at their outlet with at least two means (7, 11) which are

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mounted in series, connected to an open tank (8) and (12) respectively and using the Venturi effect, for introduction respectively at least of the emulsifying agent in said coherent jet (5) in order to form a resulting coherent jet (9) and of the continuous phase in said resulting coherent jet (9) in order to form a final coherent jet (13) and thus procure the emulsion which is continuously or discontinuously recovered at the outlet of the second introduction means (11) by drawing-off means (10).

10        8 - Emulsifier device according to either one of Claims 6 and 7, characterised in that the means (3) for connection between the high-pressure pump (2) and the spray means (4) are provided with temperature regulating means (6) over all or part of their length.

15        9 - Emulsifier device according to Claim 8, characterised in that the temperature regulation means (6) are constituted by a temperature probe (20) positioned on the connection means (3) and by a coil (18) formed by turns, connected to a heat exchanger (13), which surrounds  
20 said connection means (3).

      10 - Emulsifier device according to any one of Claims 6 to 9, characterised in that the drawing-off means (10) are provided with temperature regulation means (33, 36, 37, 38, 39) connected to the heat  
25 exchanger (13).

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3. Method according to ~~either one of the preceding Claims~~ claim 1, characterised in that the temperature of the dispersed phase under pressure is regulated in a temperature range included between -20°C and +80°C.

4. Method according to ~~any one of the preceding Claims~~ claim 1, characterised in that the contacting of the resulting coherent jet (9) with the continuous phase is obtained by positioning said resulting coherent jet (9) in immersion in the continuous phase in static or virtually static position.

5. Method according to ~~any one of Claims 1 to 4~~ claim 1, characterised in that the contacting of the resulting coherent jet (9) with the continuous phase is obtained thanks to means ensuring introduction of the continuous phase in said resulting coherent jet (9) and simultaneously their emulsion which in that case constitutes a final coherent jet (13).

8. Emulsifier device according to ~~either one of Claims 6 and 7~~ claim 6, characterised in that the means (3) for connection between the high-pressure pump (2) and the spray means (4) are provided with temperature regulating means (6) over all or part of their length.

10. Emulsifier device according to ~~any one of Claims 6 to 9~~ claim 6, characterised in that the drawing-off means (10) are provided with temperature regulation means (33, 36, 37, 38, 39) connected to the heat exchanger (13).



## ABSTRACT OF THE DISCLOSURE

A method and an implementing device, for making a mixture or an emulsion from at least an emulsifier and at least two fluids known to be immiscible, the fluids defining a dispersed phase and a continuous phase. The method is characterised in that the dispersed phase being either contained in an adapted tank, or continuously supplied, it comprises a first step which consists in pressurising the dispersed phase; then in suddenly depressurising the dispersed phase using elements generating a coherent jet (5). Then an appropriate emulsifier is introduced into the coherent jet (5) with elements for mixing the dispersed phase with the emulsifier thereby providing a resulting coherent jet (9) which is finally contacted with the continuous phase to obtain the mixture or the emulsion.

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## METHOD FOR MAKING EMULSIONS AND IMPLEMENTING DEVICE

The present invention relates to a method for making emulsions and to an emulsifier carrying out this method. Such a method will find numerous applications, particularly in the domains of cosmetology, the food industry for making French dressing for example, pharmacy, petrochemistry, etc...

The making of an emulsion generally consists in the mixture of two fluids, i.e. of two liquids, determining two phases, by hypothesis immiscible, one being called dispersed phase and the other continuous phase, and of which one forms microscopic droplets in the other. This mixture or emulsion, and more particularly the size of the droplets of the dispersed phase in the continuous phase, depends in particular on the energy furnished in the form of stirring to the medium which provokes shear of the fluid and thus allows reduction of the size of the droplets of the emulsion.

Furthermore, it is often necessary to add an emulsifying agent in order to stabilise the emulsion in time, avoiding coalescence of the dispersed phase and thus to allow storage of the emulsion. In effect, in an emulsion of the oil-in-water type where the water corresponds to the continuous phase and the oil to the dispersed phase, oil and water not being miscible, the droplets of oil will tend to collect together to form larger droplets, thus creating a phenomenon of coalescence.

Particularly in the domain of the food industry, emulsifiers are well known, such as high-pressure homogenisers or "microfluidisers" producing emulsions comprising an emulsifying agent, for example an emulsion of the oil-in-water type.

Homogenisers are conventionally constituted by a homogenisation head and a high-pressure pump in order to pressurise a fluid contained in a tank. The fluid under pressure is usually a pre-emulsion, i.e. it is question of a partial mixture of the dispersed phase, of the continuous phase and of the emulsifying agent; this fluid is then sent

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through the homogenisation head principally constituted by a base, a valve and impact plates. The pressure of the fluid is suddenly relaxed through an appropriate opening in order to attain a speed of the order of several hundreds of metres per second, then comes into contact with the valve which splits the fluid and sprays it onto the impact plates thus furnishing the necessary energy to the medium, in the form of stirring, for making the emulsion. These homogenisers, benefiting from present-day technologies, operate at pressures that may attain 200 MPa.

Such homogenisers present the major drawbacks of wear of the homogenisation head due to the considerable frictions of the fluid on the valve and the impact plates as well as a heating of the emulsion. Furthermore, for these devices operating from a pre-emulsion, a method of pre-emulsion is necessary upstream of the homogenisers, thus increasing the production costs.

In this respect, homogenisation heads have been designed, which significantly reduce their wear; this is the case for example of French Patent FR 2748954 concerning a homogenise-emulsifier module. This module is principally constituted by a cylindrical body presenting at each of its ends respectively a direct inlet unit and an outlet unit. The cylindrical body contains a succession of hollow cylindrical cartridges open on one of their transverse faces and they are connected together by springs. These cartridges contain a plurality of vibrating discs which may slide along the central hollow axis of the cylindrical body of the module. When a fluid under pressure is introduced via the direct inlet unit into the cylindrical body, all the vibrating discs start to move, thus creating an effect of shear of the fluid which allows reduction of the size of the drops of the emulsion.

"Microfluidisers" also exist which are conventionally constituted by an interaction chamber and a high-pressure pump to pressurise a fluid contained in an appropriate tank. The pressurised fluid is usually a pre-emulsion which is sent into the interaction chamber in which the latter is

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bombarded by itself with a high energy contributed by the pressurisation of the fluid, which allows the emulsion to be made.

One drawback of all these devices is that of procuring an emulsion of which the droplets present a mean diameter of the order of a micrometer, which is not fully satisfactory for applications in the food and cosmetology domains, for example.

Another drawback of these devices is the considerable quantity of emulsifying agent necessary for stabilising such an emulsion. This considerable addition of emulsifying agent is in that case translated by an excess of said emulsifying agent in the continuous phase of the emulsion after it has been made, which affects the organoleptic qualities of the emulsion in particular and increases the production costs.

One of the objects of the invention is therefore to overcome these drawbacks by proposing a method for making a mixture or an emulsion, for example of the oil-in-water type, in order to obtain a greater fineness of the droplets, using a minimum quantity of emulsifying agent for stabilising said emulsion in time.

To that end and in accordance with the invention, the method for continuously or discontinuously making a mixture or an emulsion from at least one emulsifying agent and at least two fluids known to be immiscible, for example a liquid fat mixed with water and an appropriate emulsifying agent, said fluids defining a dispersed phase and a continuous phase, is noteworthy in that, the dispersed phase being either contained in an adapted tank or continuously supplied, it comprises a first step of pressurisation of the dispersed phase by conventional high-pressure pumping means, then a sudden depressurisation of said dispersed phase is effected using means generating a needle jet, i.e. a jet of narrow cross-section, or coherent jet in which the dispersed phase may attain a speed of about  $900 \text{ m.s}^{-1}$ . In that case it can be envisaged to introduce the coherent jet of the dispersed phase in a

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continuous phase in which an appropriate emulsifying agent has been dissolved in order to obtain the emulsion.

Such a method does not make it possible to obtain a sufficiently small mean size of the droplets, and this is why it is preferred to introduce the appropriate emulsifying agent into said coherent jet thanks to means ensuring mixture of the dispersed phase with said emulsifying agent. In that case a resulting coherent jet is obtained which comprises the dispersed phase and the emulsifying agent. This resulting coherent jet is finally contacted with the continuous phase in order to obtain the mixture or the emulsion.

An emulsion is thus obtained whose droplets present a mean diameter included between some tens and some hundreds of nanometres, depending on the fluids used, while requiring a reduced addition of emulsifying agent contrarily to the prior art where the diameter of the droplets decreasing, i.e. their total surface increasing, a greater quantity of additive would have been necessary.

Furthermore, the contacting of the resulting coherent jet with the continuous phase, according to a first variant of the method, is obtained by positioning said resulting coherent jet in immersion in the continuous phase in static or quasi-static position in drawing-off means.

According to a second variant of the method, the contacting of the resulting coherent jet with the continuous phase is obtained thanks to means ensuring introduction of the continuous phase in said resulting coherent jet and simultaneously their emulsion which in that case constitutes a final coherent jet.

Upon the sudden depressurisation of the dispersed phase, the latter undergoes a heating which may in particular modify its hydrodynamic and organoleptic characteristics, and this is why the temperature of the dispersed phase under pressure is regulated in a temperature range included between -20°C and +80°C for the making of the emulsion to be more homogeneous in time.

Moreover, the dispersed phase is pressurised at a

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pressure greater than or equal to 200 MPa.

Another object of the invention concerns an emulsifier device for continuously or discontinuously making a mixture or an emulsion from at least one emulsifying agent and at least two fluids known to be immiscible, for example a liquid fat mixed with water and an emulsifying agent, said fluids defining a dispersed phase and a continuous phase, and said device comprising a high-pressure pump of which the inlet is connected to a source of fluid such as a tank containing a dispersed phase; this device is noteworthy in that the outlet of the high-pressure pump is connected, by connection means, to means for spraying the dispersed phase in the form of a coherent jet co-operating with means for introduction, using the Venturi effect, of an emulsifying agent in said coherent jet opening out, in immersion, in the continuous phase contained in a tank provided with means for drawing off the emulsion continuously or discontinuously.

According to a variant embodiment of the device comprising a high-pressure pump of which the inlet is connected to a source of fluid such as a tank containing a dispersed phase, the outlet of the high-pressure pump is connected, by connection means, to means for spraying the dispersed phase in the form of a coherent jet, provided at their outlet with at least two means, which are mounted in series and using the Venturi effect, for introduction respectively at least of the emulsifying agent in said coherent jet and of the continuous phase in the resulting coherent jet, to procure the emulsion which is advantageously continuously recovered at the outlet of said introduction means.

According to a secondary characteristic of the devices according to the invention, the connection means, between the high-pressure pump and the spray means, are provided with temperature regulating means over all or part of their length.

Other advantages and characteristics will appear more readily from the following description of several variant

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embodiments, given by way of non-limiting examples, of the method and the emulsifier device implementing it according to the invention, with reference to the accompanying drawings, in which:

5       - Figure 1 schematically shows the emulsifier device according to the invention,

      - Figure 2 is a partial diagram, in slight perspective, of the emulsifier device according to the invention comprising the tank of dispersed phase, the  
10   high-pressure pump, the connection means and the temperature regulating means,

      - Figure 3 is a partial diagram of the first variant embodiment of the emulsifier device according to the invention comprising the means for spraying the dispersed  
15   phase, the means for introducing the emulsifying agent in the jet and the drawing-off means,

      - Figure 4 is a partial diagram of the second variant embodiment of the emulsifier device according to the invention comprising the means for spraying the dispersed  
20   phase, two means for respectively introducing the emulsifying agent and the continuous phase mounted in series, and the drawing-off means,

      - Figure 5 is a graph representing the percentage (%) of the droplets as a function of their diameter expressed  
25   in nanometres (nm) for an example of emulsion of the oil-in-water type, comprising 10% of sunflower oil, 89% of water and 1% of emulsifying agent Tween 20 (Registered Trademark), and obtained by spraying a jet of sunflower oil, pressurised at 200 MPa, in water in which the Tween 20  
30   (Registered Trademark) was previously dissolved,

      - Figure 6 is a graph representing the percentage (%) of the droplets as a function of their diameter expressed in nanometres (nm) for an emulsion of the oil-in-water type, comprising 10% of sunflower oil,  
35   89.5% of water and 0.5% of emulsifying agent Tween 20 (Registered Trademark) and obtained according to the method,

      - Figure 7 is a graph representing the influence of

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the emulsifying/continuous phase ratio on the stability of an emulsion of the water-in-oil type.

For reasons of clarity, "emulsion" will designate hereinafter all mixtures and emulsions obtained according to the invention and "emulsifier" all the mixer, homogeniser, "microfluidiser", emulsifier and homogeniser-emulsifier devices.

The device for continuously or discontinuously making an emulsion which is shown in Figures 1 to 4, comprises a tank 1 containing a dispersed phase and of which the outlet is connected to a high-pressure pump 2. A booster pump (not shown in the Figures) will advantageously be positioned between the tank 1 and the high-pressure pump 2 in order to prime the latter in conventional manner. The outlet of the high-pressure pump 2 is connected, with reference to Figure 1, by connection means 3, to means 4 for spraying the continuous phase in the form of a needle jet or coherent jet 5. Furthermore, the connection means 3, between the high-pressure pump 2 and the spray means 4, are provided with means 6 for regulating the temperature of the dispersed phase, under pressure in said connection means 3, over the whole or part of their length. The outlet of the spray means 4 is provided with means 7 for introducing in the coherent jet 5 an emulsifying agent contained in a second tank 8 connected to said introduction means 7 so that a resulting coherent jet 9, constituted by the dispersed phase and the emulsifying agent, sprays at their outlet. The resulting coherent jet 9 is then contacted with the continuous phase contained in continuous or discontinuous drawing-off means 10 as will be seen hereinbelow. The resulting coherent jet 9 is preferably positioned in immersion in said continuous phase in order to benefit from the optimum energy, of said resulting coherent jet, necessary for obtaining a fine emulsion.

According to a variant embodiment of the emulsifier device according to the invention, the outlet of the introduction means 7 is provided with second introduction means 11, shown in dotted lines in Figure 1, in the



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resulting coherent jet of a continuous phase contained in a third tank 12, also shown in dotted lines in Figure 1, connected to said introduction means 11 so that a final coherent jet 13 constituted by the emulsion sprays at their outlet. The final coherent jet 13, i.e. the emulsion, is then continuously or discontinuously collected in the drawing-off means 10.

With reference to Figure 2, the tank 1, containing the dispersed phase, is connected to the high-pressure pump 2 by a pipe 14. The high-pressure pump 2 is advantageously a return pump which presents a very short time constant and which therefore does not present any idle time. It makes it possible to obtain a pressure of 400 MPa while ensuring a high output and constant pressure. The means 3 for connection between the high-pressure pump 2 and the spray means 4 (not shown in Figure 2) are constituted by an armoured pipe 15 adapted to convey the pressurised dispersed phase and they present a branch circuit 16 provided with control valves 17 such as electro-valves. The branch circuit 16 comprises means 6 for regulating the temperature of the pressurised dispersed phase shown in dotted lines in Figure 2. The regulation means 6 are, furthermore, constituted by a coil 18 formed by turns surrounding the armoured pipe 15 on a part of the branch circuit 16 and connected to a heat exchanger 19.

It goes without saying that the length of the coil 18 depends in particular on the heat coefficients of the calorific fluid circulating in said coil 18 and on the dispersed phase used. Moreover, the connection means 3 do not necessarily comprise a branch circuit 16 and the coil 18 will in that case be directly positioned around the armoured pipe 15.

Furthermore, the regulation means 6 also comprise a probe 20, preferably mounted upstream of the coil 18 on the branch circuit 16, making it possible to monitor the temperature of the dispersed phase in the armoured pipe 15.

In accordance with a first variant of the emulsifier device according to the invention shown in Figure 3, the

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spray means 4 are conventionally mounted at the end of the armoured pipe 15, facing the ground and they are constituted by a nozzle 21 supported by a nozzle-holder 22 comprising a calibrated hole 23. The nozzle 21 is conventionally constituted by a body 24 comprising at its lower end a second calibrated hole 25 and by a needle 26 comprising a third calibrated hole 27 coaxial to the first 23 and to the second 25. The diameter of the calibrated hole 26 is advantageously included between 0.08 and 0.15 mm for a pressure delivered by the high-pressure pump 2 of 200 MPa in order to avoid said calibrated hole 26 being obstructed.

It goes without saying that the spray means 4 may be directed upwardly in order to procure a straight jet.

The nozzle 21 procures a needle jet, i.e. a jet of narrow cross-section, or coherent jet 5 of the dispersed phase which is suddenly depressurised and which sprays into the introduction means 7. Said introduction means 7 are positioned at the lower end of the nozzle-holder 22 and are constituted by a Venturi tube 28, of a length of about 15 mm for a pressure included between 200 MPa and 300 MPa, forming in its central part a mixing chamber 29 and at its lower end a focussing tube 30. The coherent jet 5 thus sprays into the mixing chamber 29 where the emulsifying agent, initially contained in the tank 8 and which is conducted, by a flexible pipe 31 provided with a control valve 17 and a flowrate regulation system 32, into the mixing chamber 29 by Venturi effect, mixes in order to procure in the focussing tube 30 a resulting coherent jet 9.

It should be noted that the tank 8 is an open tank so that the emulsifying agent is at atmospheric pressure and can benefit from the Venturi effect in order to be conducted into the mixing chamber 29. Furthermore, it could be envisaged to introduce the emulsifying agent in the coherent jet of the dispersed phase by means of an incidental jet making a very small angle with said coherent jet 5.

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The focussing tube 30 is positioned in immersion in a static or virtually static continuous phase contained in the drawing-off means 10 which are constituted by a principal cylindrical recipient 33, a median cylindrical recipient 34 and a central cylinder 35, all coaxial. The principal cylindrical recipient 33 presents the largest cross-section and comprises two openings 36, 37 in its upper part for the introduction of a calorific fluid and two other openings 38, 39 in its lower part for the outlet of said calorific fluid, as will be seen hereinbelow. The openings 36, 37, 38 and 39 of the principal cylindrical recipient 33 are advantageously connected to the heat exchanger 19 by conventional connection means (not shown in the Figures). The median cylindrical recipient 34, positioned inside the principal cylindrical recipient 33, comprises a reinforced bottom 40 to avoid deformation thereof due to the pressure of the resulting coherent jet 9. The central cylinder 35, opening at its two ends, is positioned in the median cylindrical recipient 34 so that its lower end 41 is not in contact with the reinforced bottom 40. Furthermore, the median cylindrical recipient 34 and the central cylinder 35 respectively comprise an opening 42 in its central part for drawing-off of the emulsion and an opening 43 in its upper part for the introduction of the continuous phase, as will be seen hereinbelow.

It goes without saying that the drawing-off means 10 may be constituted by a single cylindrical recipient comprising the dispersed phase and provided, or not, with an opening in its upper part for the introduction of the continuous phase and with another opening in its lower part for drawing off the emulsion either continuously or discontinuously.

According to a second variant of the emulsifier device according to the invention, shown in Figure 4, the spray means 4, as described hereinabove, procure a coherent jet 5 which sprays into a first Venturi tube 28 as described hereinabove allowing the mixture of the

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emulsifying agent, previously contained in the tank 8, with the dispersed phase and procuring a resulting coherent jet 9 as has already been seen. Said resulting coherent jet 9 in that case sprays into a second Venturi tube 44 mounted in series with the first 28 and forming a second mixing chamber 45 in its central part and a second focussing tube 46 in its lower part. The resulting coherent jet 9 thus sprays into the second mixing chamber 45 where the continuous phase, initially contained in the tank 12 then conducted, by a flexible conduit 31 provided with a control valve 17 and a flowrate regulation system 32, into the second mixing chamber 45 by Venturi effect, mixes with said resulting coherent jet 9 to procure the emulsion which flows in the second focussing tube 46 in the form of a final coherent jet 13.

It goes without saying that the device may comprise a plurality of Venturi tubes mounted in series making it possible to introduce successively in the coherent jet 5 a plurality of emulsifying agents and a plurality of continuous phases in order to make so-called ternary emulsions such as emulsions of the water-oil-water type.

The final coherent jet 13, i.e. the emulsion, is collected in the drawing-off means 10 placed vertically under the second focussing tube 46. The drawing-off means 10 are in that case constituted by a simple cylindrical recipient 47 provided with an opening 48 in its lower part to continuously draw off the emulsion as indicated by arrow 49.

Naturally, the emulsion might be drawn off discontinuously by using a simple cylindrical recipient.

The functioning of the emulsifier device according to the invention will now be explained with reference to Figures 2, 3, 5 and 6. To make an emulsion of oil-in-water type for example, sunflower oil, which in this case will correspond to the dispersed phase, is placed in the tank 1; then, by means of a booster pump (not shown in Figure 2), the high-pressure pump 2 is primed, which then pressurises the oil in the armoured pipe 15. If necessary, the

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different control valves 17 are then actuated for the oil to circulate in the branch circuit 16 in order to regulate its temperature. The pressurised oil sprays from the nozzle 21 (Figure 3) in order to form a coherent jet 5 through the Venturi tube 28. The oil is preferably pressurised at a pressure greater than or equal to 200 MPa so that the coherent jet 5 has sufficient energy to form the emulsion without the nozzle 21 being obstructed. The speed of the oil may then attain  $900 \text{ m.s}^{-1}$  for a pressure of 200 MPa and a diameter of the nozzle 21 included between 0.08 and 0.15 mm.

For reasons of clarity, Tween 20 will designate the emulsifying agent used, Tween 20 being a Registered Trademark for an emulsifying agent which will be called "Tween 20" hereinafter.

By Venturi effect, the "Tween 20" is sucked by the coherent jet 5 of oil with which it is mixed in order to form the resulting coherent jet 9.

It should be noted that the "Tween 20" does not dissolve in the dispersed phase, i.e. the oil. Generally, the emulsifying agent only dissolves in the continuous phase; thus, the "Tween 20" mixes homogeneously in the coherent jet 5 but without being dissolved therein.

The resulting coherent jet 9 is then introduced in immersion in the water, corresponding to the continuous phase, which is continuously injected in the central cylinder 35 via the opening 43 as indicated by arrow 50 in Figure 3.

When the resulting coherent jet 9 which consists of the mixture of the oil and of the "Tween 20" comes into contact with the water, droplets of oils form in the water and the "Tween 20" is positioned around these droplets to avoid their collecting together and an emulsion of the oil-in-water type is thus obtained. The emulsion obtained in this way continues to descend in the central cylinder 35, thereafter rising between the walls of the median cylindrical recipient 34 and said central cylinder 35, as indicated by arrows 51, and finally drawn off through the

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opening 42, as indicated by arrow 52. The temperature of the emulsion may then be regulated thanks to the passage of a calorific fluid between the principal cylindrical recipient 33 and the median cylindrical recipient 34. The calorific fluid enters via the upper openings 36, 37, as indicated by arrows 53 and emerges via the lower openings 38, 39 as indicated by arrows 54 in Figure 3.

The size of the droplets of the emulsion, and more precisely their diameter, depends in particular on the energy contributed in the form of stirring to the medium as has already been seen, but also on the fluids used. For an emulsion of the oil-in-water type for example, the size of the droplets will depend in particular on the type of oil used.

Figure 5 represents the percentage of the droplets as a function of their diameter, expressed in nanometres (nm) for an emulsion of the oil-in-water type, comprising 10% of sunflower oil, 89% of water and 1% of emulsifying agent "Tween 20", and obtained by spraying a jet of sunflower oil, pressurised at 200 MPa, in water in which the "Tween 20" was previously dissolved. The general shape of the curve as well as the peak around 500 nm indicate that the mean diameter of the droplets of the emulsion is included between 500 and 600 nm. For an emulsion comprising 10% of sunflower oil, 89.5% of water and 0.5% of emulsifying agent "Tween 20" and obtained according to the invention, the percentage of the droplets as a function of their diameter, expressed in nanometres, represented in Figure 6, presents a different curve. In effect, a first peak is observed at about 200 nm and a second peak at around 450 nm indicating a more rapid stabilisation of the dispersed phase, i.e. of the oil, then a slight phenomenon of coalescence. An emulsion is therefore obtained of which the size of the droplets is smaller for a lesser quantity of emulsifying agent, as specified in Figure 7.

The graph of Figure 7 represents the destabilisation expressed in percentage, on the y-axis, which corresponds to the percentage of the quantity of the destabilised phase

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with respect to its initial quantity, as a function of the emulsifying agent/dispersed phase ratio, on the x-axis, i.e. the ratio of the percentages of emulsifying agent and of dispersed phase of the emulsion. The curve in dotted lines corresponds to an emulsion obtained by introducing a jet of water, pressurised at 200 MPa, into oil in which the "Tween 20" was previously introduced and the solid-line curve corresponds to an emulsion obtained according to the invention. With reference to Figure 7, it is observed that the stabilisation, i.e. a zero destabilisation, is obtained at a ratio of about 0.03 for an emulsion according to the invention and at a ratio of about 0.12 for the other emulsion obtained conventionally. Consequently, a smaller quantity of emulsifying agent is necessary in order to stabilise the emulsion. In effect, it may be reasonably estimated that the "Tween 20" being introduced homogeneously into the oil only a few milliseconds before the impact with the water, due to the dimensions of the Venturi tube 28 and to the speed of the oil in the coherent jet 5, only the emulsifying agent necessary for stabilisation of the interface of the dispersed and continuous phases is required and therefore added.

It is obvious that the values plotted in Figures 5, 6 and 7 are purely indicative and vary as a function of the types of emulsion. Furthermore, depending on the types of emulsion and their applications, an appropriate emulsifying agent will be used.

Finally, it goes without saying that the method according to the invention and the emulsifier device implementing it allow all types of emulsion to be made, particularly emulsions of the water-in-oil type or of the ternary type, and the examples which have just been given are only particular illustrations which are in no way limiting as to the domains of application of the invention.

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## CLAIMS

1 - Method of continuously or discontinuously making a mixture or an emulsion from at least one emulsifying agent and at least two fluids known to be immiscible, for example a liquid fat mixed with water and an appropriate emulsifying agent, said fluids defining a dispersed phase and a continuous phase, characterised in that, the dispersed phase being either contained in an adapted tank or continuously supplied, at least the following steps are carried out in order:

10       - the dispersed phase is pressurised by conventional high-pressure pumping means, then

      - the dispersed phase is suddenly depressurised using means generating a coherent jet (5) then

15       - an appropriate emulsifying agent is then introduced into said coherent jet (5) thanks to means ensuring mixing of the dispersed phase with said emulsifying agent and thus procuring a resulting coherent jet (9) then

      - said resulting coherent jet (9) is contacted with the continuous phase in order finally to obtain the emulsion.

2 - Method according to Claim 1, characterised in that the fluid or fluids forming the dispersed phase is pressurised at a pressure greater than or equal to 200 MPa.

25       3 - Method according to either one of the preceding Claims, characterised in that the temperature of the dispersed phase under pressure is regulated in a temperature range included between -20°C and +80°C.

30       4 - Method according to any one of the preceding Claims, characterised in that the contacting of the resulting coherent jet (9) with the continuous phase is obtained by positioning said resulting coherent jet (9) in immersion in the continuous phase in static or virtually static position.

35       5 - Method according to any one of Claims 1 to 4, characterised in that the contacting of the resulting coherent jet (9) with the continuous phase is obtained thanks to means ensuring introduction of the continuous



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phase in said resulting coherent jet (9) and simultaneously their emulsion which in that case constitutes a final coherent jet (13).

5 6 - Device for continuously or discontinuously making a mixture or an emulsion from at least one emulsifying agent and at least two fluids known to be immiscible, for example a liquid fat mixed with water and an appropriate emulsifying agent, said fluids defining a dispersed phase and a continuous phase, implementing the method according to Claim 4 and comprising a high-pressure pump (2) of which  
10 the inlet is connected to a source of fluid such as a tank (1) containing a dispersed phase, characterised in that the outlet of the high-pressure pump (2) is connected, by connection means (6), to means (4) for spraying the  
15 dispersed phase in the form of a coherent jet (5) co-operating with means (7), connected to a tank (8) and using the Venturi effect, for introduction of the emulsifying agent in said coherent jet (5) to form a resulting coherent jet (9) opening out, in immersion, in the continuous phase  
20 contained in means (10) for drawing off the emulsion continuously or discontinuously.

7 - Device for continuously or discontinuously making a mixture or an emulsion from at least one additive and at least two fluids known to be immiscible, for example a  
25 liquid fat mixed with water and an appropriate emulsifying agent, said fluids defining a dispersed phase and a continuous phase, implementing the method according to Claim 5 and comprising a high-pressure pump (2) of which the inlet is connected to a source of fluid such as a  
30 tank (1) containing a dispersed phase, characterised in that the outlet of the high-pressure pump (2) is connected, by connection means (3), to means (4) for spraying the dispersed phase in the form of a coherent jet (5), provided at their outlet with at least two means (7, 11) which are  
35 mounted in series, connected to a tank (8) and (12) respectively and using the Venturi effect, for introduction respectively at least of the emulsifying agent in said coherent jet (5) in order to form a resulting coherent

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jet (9) and of the continuous phase in said resulting coherent jet (9) in order to form a final coherent jet (13) and thus procure the emulsion which is continuously or discontinuously recovered at the outlet of the second introduction means (11) by drawing-off means (10).

8 - Emulsifier device according to either one of Claims 6 and 7, characterised in that the means (3) for connection between the high-pressure pump (2) and the spray means (4) are provided with temperature regulating means (6) over all or part of their length.

9 - Emulsifier device according to Claim 8, characterised in that the temperature regulation means (6) are constituted by a temperature probe (20) positioned on the connection means (3) and by a coil (18) formed by turns, connected to a heat exchanger (13), which surrounds said connection means (3).

10 - Emulsifier device according to any one of Claims 6 to 9, characterised in that the drawing-off means (10) are provided with temperature regulation means (33, 36, 37, 38, 39) connected to the heat exchanger (13)

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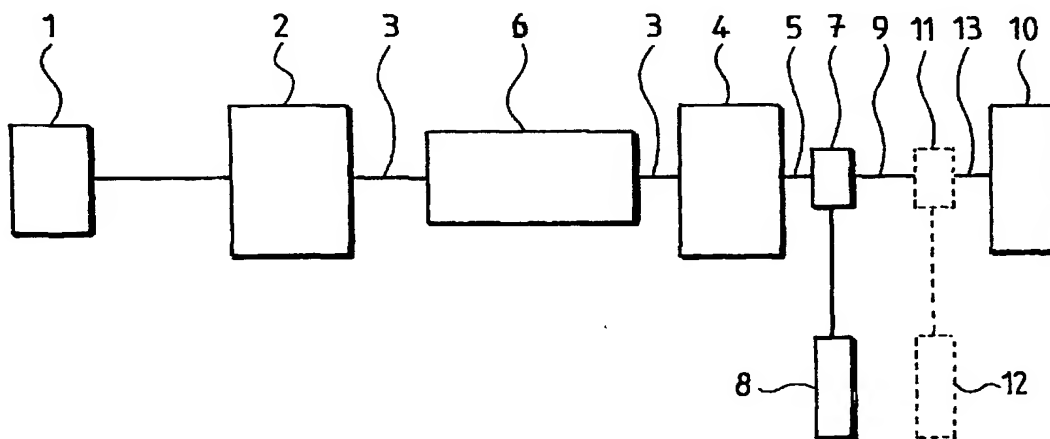
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[Suite sur la page suivante]

(54) Title: METHOD FOR MAKING EMULSIONS AND IMPLEMENTING DEVICE

(54) Titre: PROCEDE DE FABRICATION D'EMULSIONS ET SON DISPOSITIF



(57) Abstract: The invention concerns a method and an implementing device, for making a mixture or and emulsion from at least an emulsifier and at least two fluids known to be immiscible, said fluids defining a dispersed phase and a continuous phase. The method is characterised in that the dispersed phase being either contained in an adapted tank, or continuously supplied, it comprises a first step which consists in pressurising the dispersed phase; then in suddenly depressurising said dispersed phase using means generating a coherent jet (5). Then an appropriate emulsifier is introduced into said coherent jet (5) with means for mixing the dispersed phase with said emulsifier thereby providing a resulting coherent jet (9) which is finally contacted with the continuous phase to obtain the mixture or the emulsion.

(57) Abrégé: L'invention concerne un procédé, ainsi qu'un dispositif le mettant en oeuvre, pour la fabrication d'un mélange ou d'une émulsion à partir d'au moins un émulsifiant et au moins deux fluides réputés non miscibles, lesdits fluides définissant une phase dispersée et une phase dispersante; ce procédé est remarquable en ce que, la phase dispersée étant soit contenue dans un réservoir adapté, soit délivrée en continu, il comporte une première étape

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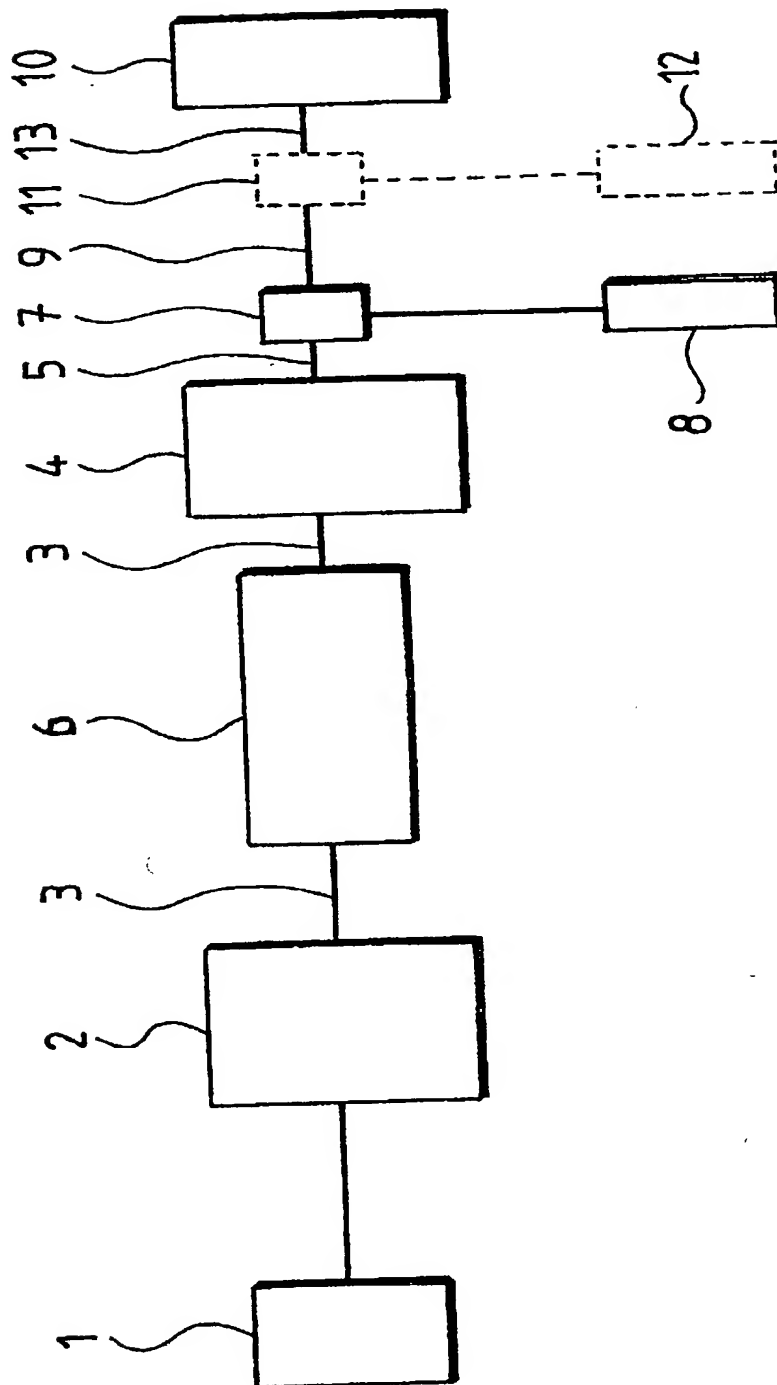
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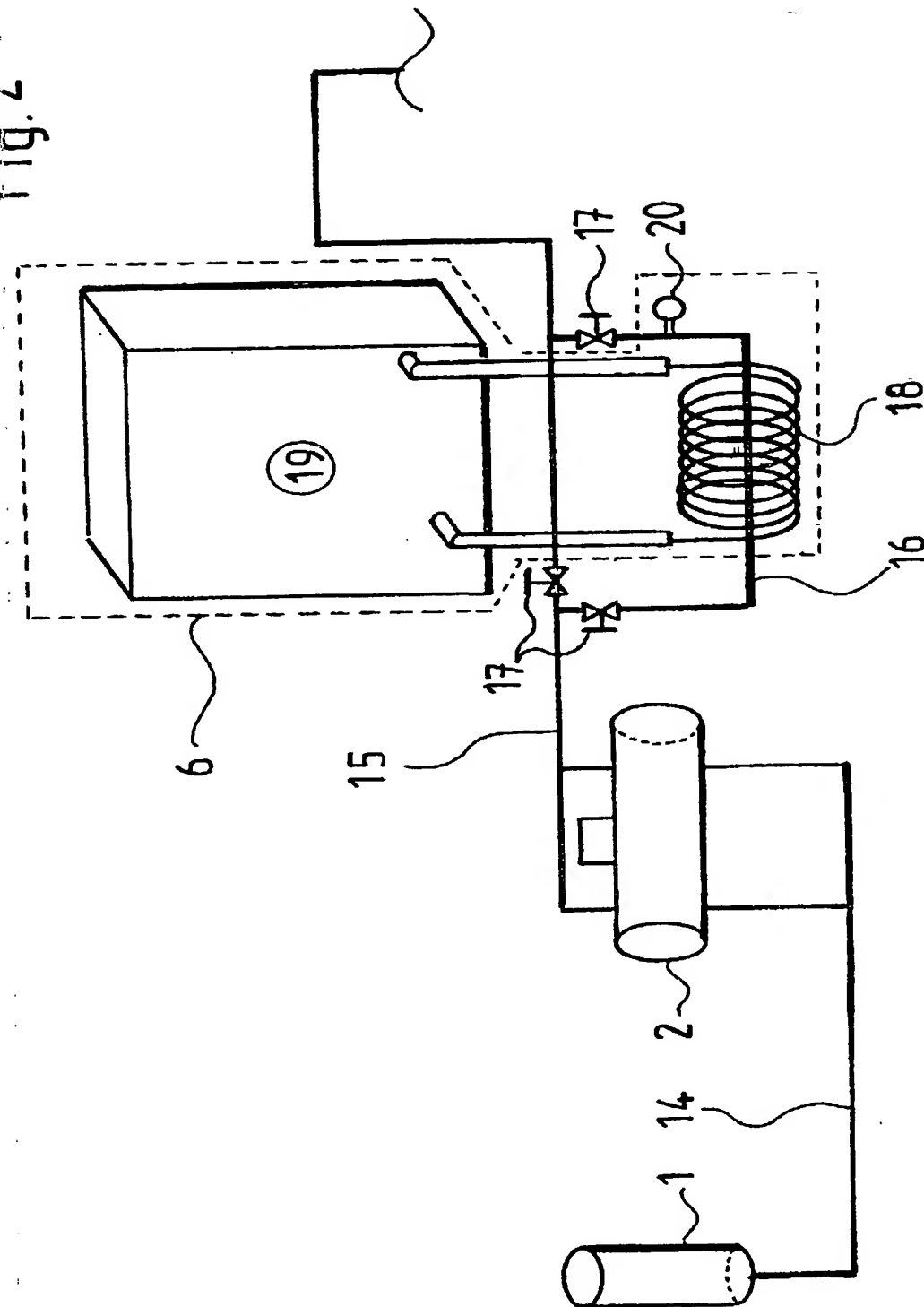
fig. 1

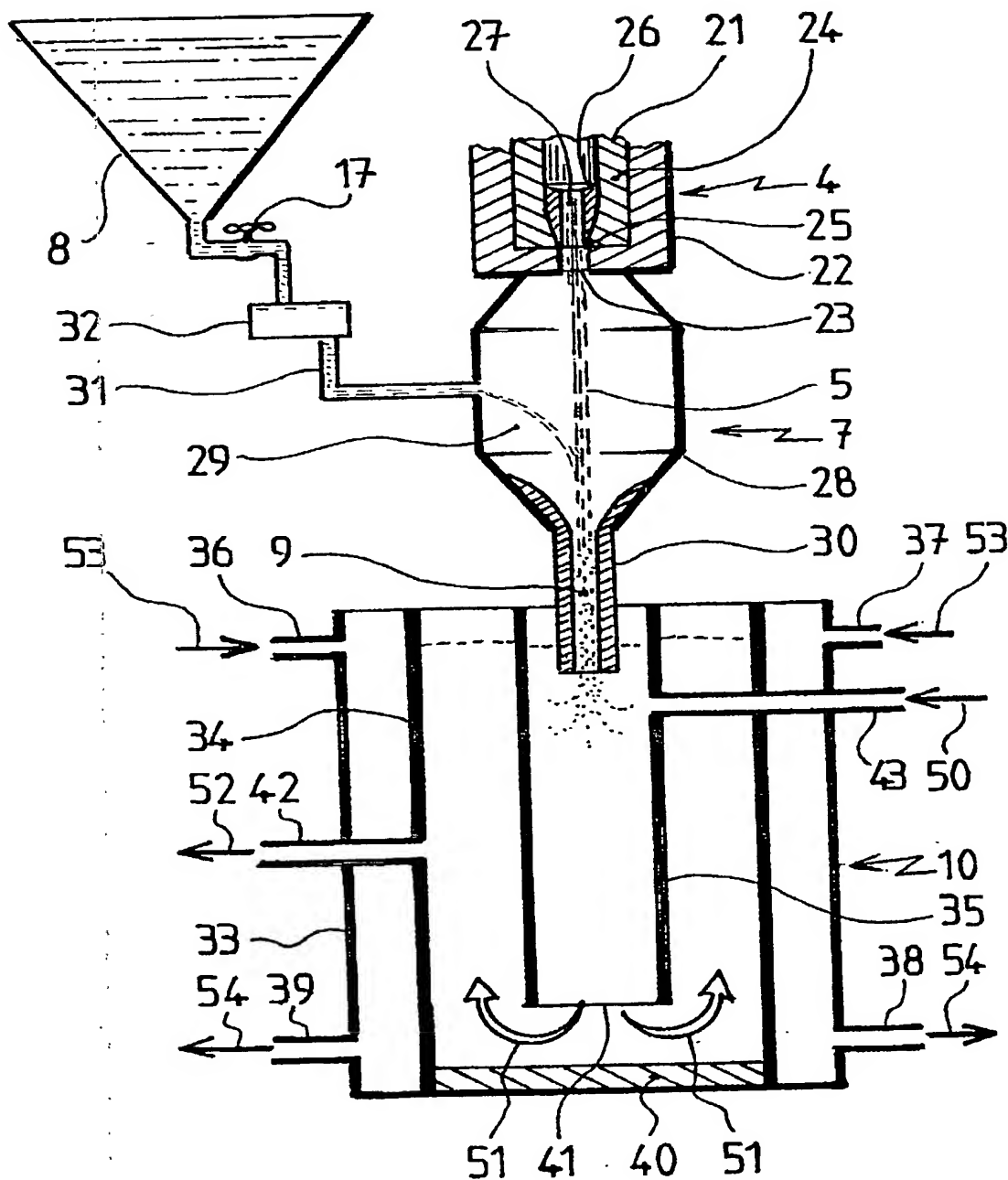


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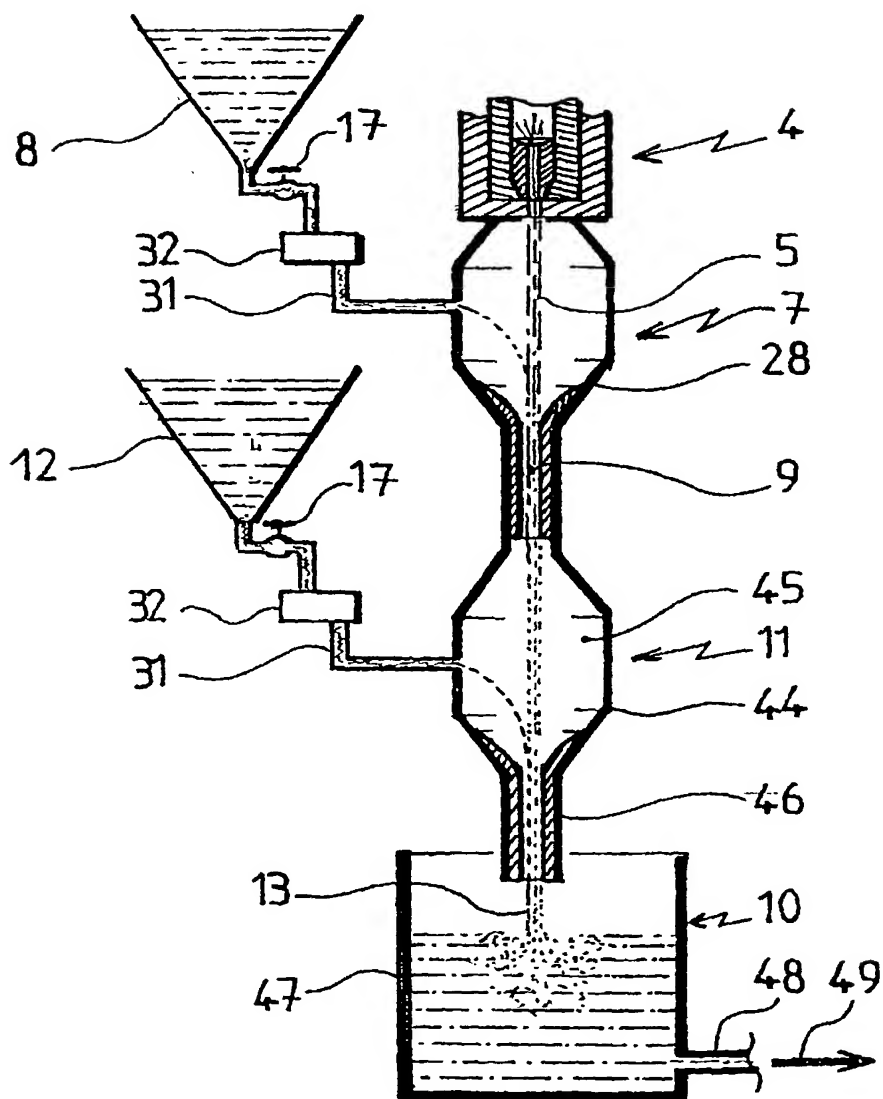
fig. 2



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fig.3

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fig. 4



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fig. 5

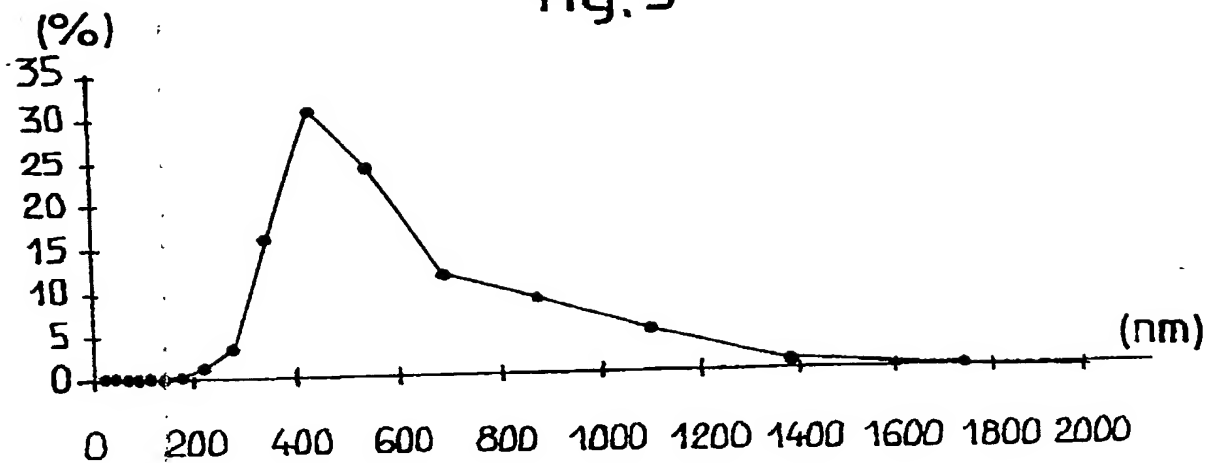
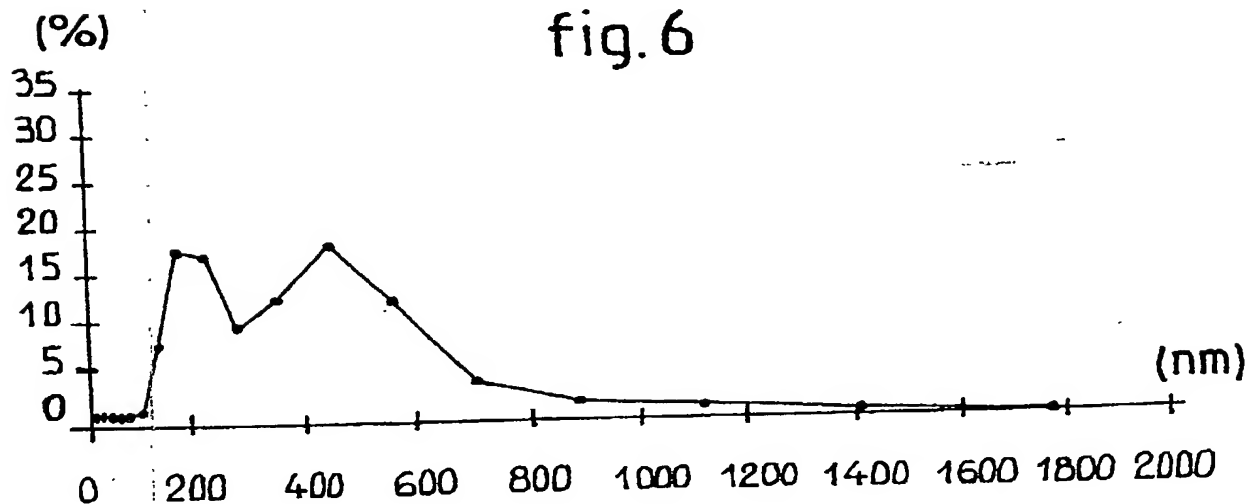


fig. 6



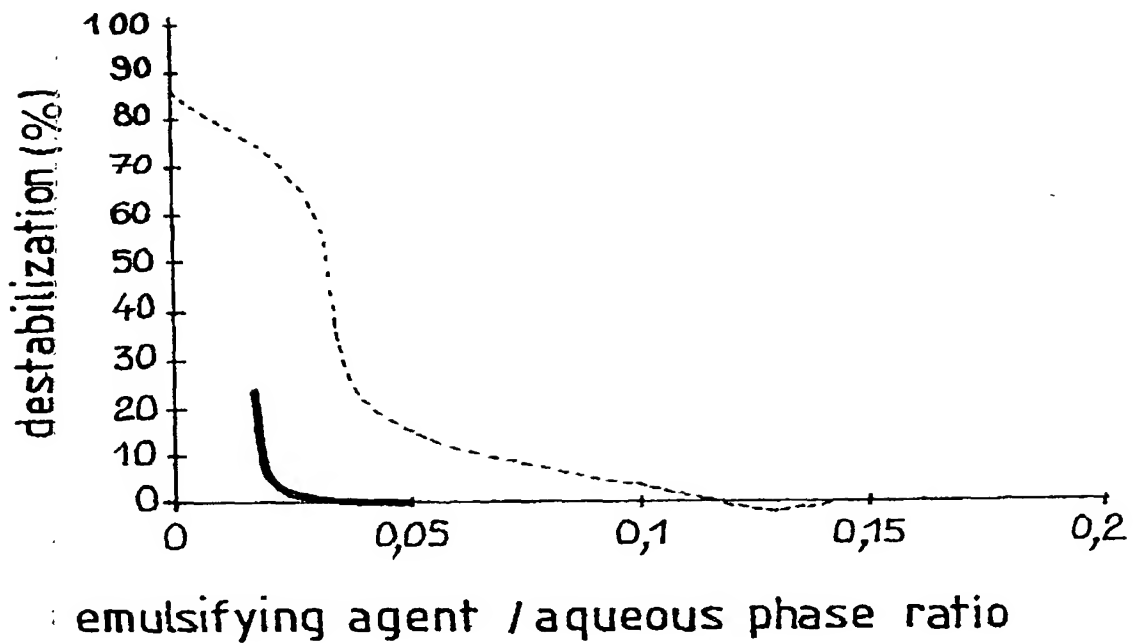


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fig.7



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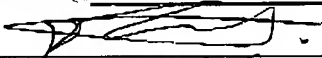
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor: **Eric FERRET**

Inventor's signature: 

Date: 15/04/02

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Inventor's signature: 

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Inventor's signature: 

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Full name of fifth joint inventor, if any: Philippe MARIE

Inventor's signature: *[Signature]*

Date:

08/04/02

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Full name of sixth joint inventor, if any: Isabelle DE LAMARLIERE

Inventor's signature: *[Signature]*

Date:

08/04/02

Residence: LONGECOURT EN PLAINE, FRANCE

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Full name of seventh joint inventor, if any: Elisabeth LUSTRAT

Inventor's signature: *[Signature]*

Date:

11/04/02

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Full name of eighth joint inventor, if any: Gilles FRANCH

Inventor's signature: *[Signature]*

Date:

08/04/02

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Full name of ninth joint inventor, if any:

Inventor's signature:

Date:

Residence:

Citizenship:

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